VIABILITY STUDY FOR THE INSTALLATION OF A SANS BEAM-LINE AT IPEN/CNEN-SP

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ABSTRACT

It is presented a study of the performance parameters of a hypothetical small angle neutron scattering (SANS) spectrometer at the IEA-R1 reactor. It is shown that the range of useful momentum transfer Q for a traditional SANS spectrometer would be 0.01 to 0.5 $Å^{-1}$ with a resolution $\Delta Q/Q$ of 70% at Q_{min}. Possibilities to improve the resolution and to decrease the lower limit of momentum transfer are discussed.

Keywords: neutron scattering, SANS

I. INTRODUCTION

The small angle neutron scattering (SANS) and the X-ray counterpart (SAXS) are techniques used to infer the structure of a material in the so-called mesoscopic scale covering the length range of 10 to 1000 Å [1]. It is nowadays in widespread use in materials science, polymer chemistry, biology and magnetism. Specifically, structure of large molecules such as colloid particles and micelles, magnetic inhomogeneities, microvoids, defect properties and precipitates can be studied with SANS and SAXS. SANS is usually considered to be complementary to SAXS and electron microscopy but problems of magnetism and some experiments on colloid science can only be studied by SANS.

A typical small angle neutron scattering facility is sketched in Fig 1. It consists mainly of the following four parts: 1) a neutron guide that transmits neutrons of low energy to places that are far apart from the reactor beam port, in order to minimize the background due to epithermal and fast neutrons as well as gamma radiation; 2) a neutron velocity selector; 3) a pinhole collimator; 4) a large area position-sensitive neutron detector. The distance between the end of the guide and the neutron detector is always higher than 10 m and in some cases, more than 100 m. Due to these large distances traveled by the neutrons in these experiments, SANS facilities usually run only at the high flux reactors.

As new materials and devices for neutron experiments became available in the last decade and new design strategies for SANS instruments appeared, the question about the possibility to run SANS instruments with the medium flux reactors was raised naturally.

The purpose of this work is to study the viability to implement a SANS facility at one beam port of the IEA-R1 reactor at IPEN, with special emphasis to determine the potentialities and the limitations. Among the motivations for this study one can cite:

- The number of potential users among the Brazilian scientific community is known to be enough to justify the investment and is still growing during the last years.
- The SAXS line from the synchrotron light laboratory at Campinas, São Paulo, is now well established and it will be attractive for regular SAXS users from this line to perform new or complementary experiments on a SANS line.
- The IEA-R1 reactor represents the unique place in Brazil where a source of neutrons is available.

II. FUNDAMENTALS OF SANS

In a scattering experiment, a pattern of scattered particles (or waves) from a sample is determined with the aim to get information about the form and spatial arrangement of the elementary components of the sample, i.e., about the structure of the sample. A collimated beam of particles (visible light, X-rays, neutrons) is made incident upon the sample and the intensity of scattered particles as a function of the scattering angle is measured. The fundamental relationship which governs the

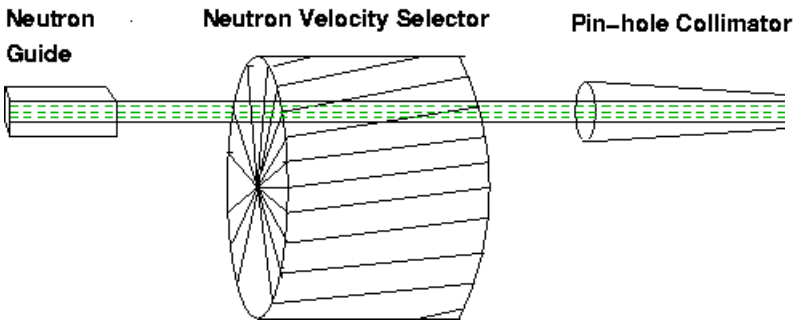
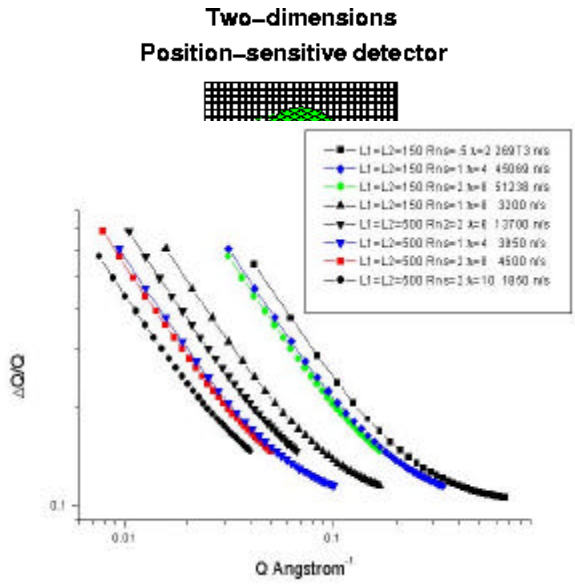


Figure 1. Sketch of a SANS ins



experiment is the one given by the so-called *scattering amplitude*:

$$f = \int dV \cdot n(\mathbf{r}) \cdot \exp(-i\mathbf{Q} \cdot \mathbf{r}) \qquad (1)$$

the square of which determines the scattered intensity. Here, the integral is extended over the volume of the sample, $n(\mathbf{r})$ is the charge (X-ray) or mass (neutron) density and **Q** is the scattering vector, given by:

$$\mathbf{Q} = \mathbf{k}_0 - \mathbf{k}_1 \qquad (2)$$

being $\mathbf{k}_0$ and $\mathbf{k}_1$ the incident and scattered wave vectors respectively. The magnitude of the scattering vector is:

$$Q = \frac{2p}{l} q \qquad (3)$$

where l is the wavelength of the beam particles and q the scattering angle. The quantities **Q** and **r** in equation 1 are reciprocally related and, as a consequence, to probe greater spatial inhomogeneity, smaller regions of Q have to be scanned or, according to 3, smaller scattering angles for fixed wavelengths.

The periodic arrangement of atoms in solids can be observed by 1 Å neutrons or X-rays scattered into the 10 to 120 degrees interval whereas within the small scattering angle range (~ 0.1º to 3º), spatial inhomogeneities with dimensions of 10 to 1000 Å is covered. Larger wavelengths are advantageous in this case and neutron wavelengths of until 20 Å have been utilized.

In order to cover such small angles, a beam of high intensity, small cross-section (~1 mm^2) and sample-to-detector distance around 1 meter are needed. These figures are usually found in experiments with X-rays in synchrotron radiation facilities. In the case of SANS, due to the much smaller beam densities, the sample-to-detector distance can reach more than 50 meters since a wider beam (~ 1cm) is needed in this case.

III. THE OPTICS OF A SANS INSTRUMENT

The performance of a SANS instrument depends basically on the magnitudes of the pinhole collimator apertures and dimension as well as the distances between sample and detector, shown in Fig. 2. The entrance and exit collimator apertures are R_{ns} and R_s respectively. L_1 is the collimator length and L_2 is the sample-to-detector distance [1]. The detector is considered to have a pixel of size Δl. Relations between these variables can be obtained in order to get the higher counting rate at the detector for a given resolution of the instrument. These are:

$$L1 = L2 \text{ and } R_{ns} = 2 \cdot R_s = \Delta l \qquad (4)$$

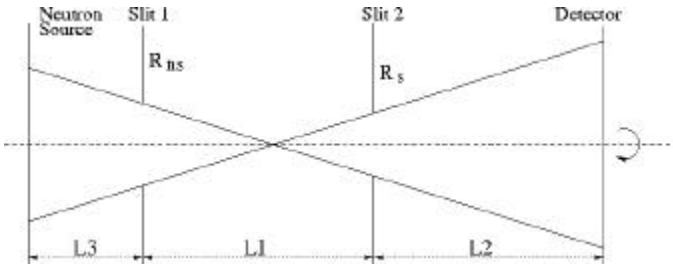


Figure 2. Neutron optics of a SANS instrument

The magnitude of the scattering vector corresponding to the *n*'th detector pixel from the center of the detector is

$$Q = \frac{2 \cdot p}{l} \cdot \frac{n \cdot \Delta l}{L_2} \qquad (5)$$

Whereas the minimum Q is given by:

$$Q_{\min} = \frac{2 \cdot p}{l} \cdot \left(2\frac{R_{ns}}{L_1} + \frac{\Delta l}{L_2} \right) \qquad (6)$$

$Q_{\max}$ is determined by (4) with $n = n_0$, the outermost pixel of the detector.

The resolution of the spectrometer and the number of neutrons hitting the sample per unit time, are respectively given by:

$$\frac{\Delta Q}{Q} = \left[\left(\frac{\Delta l}{l} \right)^2 + \left(\frac{\Delta \Theta}{\Theta} \right)^2 \right]^{1/2} \qquad (7)$$

$$N_s = \frac{\Phi_0 p}{4} \frac{{R_{ns}}^4 {L_2}^2}{(L_1 + L_2)^2 {L_1}^2} \bullet \left\{ 1 + \frac{1}{3} \frac{L_2^2 L_3^2}{(L_1 + L_2)^2 (L_1 + L_3)^2} \right\} \qquad (8)$$

where l is the neutron wavelength, $\Delta\Theta$ is the angular spread of scattered neutrons, as the full width at a half maximum (FWHM) of a gaussian distribution:

$$\Delta\Theta = \sqrt{2 \ln 2} \left[2\left(\frac{R_{ns}}{L_1} \right)^2 + \frac{1}{3}\left(\frac{\Delta l}{L_2} \right)^2 \right]^{1/2} \qquad (9)$$

and Φ_0 is the neutron flux at the neutron source.

IV. PERFORMANCE OF A SANS INSTRUMENT

Figure 3 shows the result of a simulation of the performance of an hypothetical SANS instrument running at the beam port #10 of IEA-R1 reactor. The temperature of the Maxwell distribution of thermal neutrons was estimated to be 350 K. The available space at this position is 16 m long. The length of the instrument, $L_1 + L_2$, was taken to be 10 m, being the rest of the available space supposed to be reserved to the exit portion of a stacked neutron guide, a neutron velocity selector and the sample environment. The detector is a typical 64 x 64 pixels, position sensitive in two dimensions, each pixel being 1 cm x 1 cm wide. The width of the neutron wavelengths impinging the sample was taken to be 5% around the mean wavelength.

In Fig. 3, several curves representing the instrument resolution $\Delta Q/Q$ as a function of the momentum transfer Q are presented. The parameters for each curve are shown in the insert together with the number of neutrons hitting the sample per second at a given condition.

The acceptable minimum for the neutron rate at the sample position is taken to be 10^4 neutrons per second. Taking in mind that only 10% of these are useful for the experiment in order to avoid multiple scattering and considering the losses on the neutron filter and on the monochromator that together can easily bear 70%, around 300 neutrons per second would be reliable for counting. A typical experiment with good statistics would last about one hour and more if one considers the background counting as it is expected to be high in this case since the spectrometer has to be installed in the reactor room.

With this lower limit, it can be seen that the lower Q range would be attainable with 6 Å neutrons with $L1 = L2 = 5$ m and R_{ns} = 2 cm, and, since $R_{ns} = 2R_s$,

Figure 3. Plots of the resolution FWHM as a function of the momentum transfer. In the insert the conditions for each curve are shown. Lengths are in cm, wavelength in Å. The last column represents the number of neutrons hitting the sample per second. Neutron flux at the reator core Φ_0 = 10^{12} n·cm^{-2}·s^{-1}, temperature of the Maxwell distribution T=350K

the sample would have a diameter of 2 cm. Larger wavelengths can only be utilized with larger samples and consequently deteriorating the resolution. Combining the conditions ($L_1 = L_2 = 5$ m, $l = 6$ Å) with ($L_1 = L_2 = 1.5$ m, $l = 4$ Å) a useful Q range from 0.01 Å to 0.5 Å can be envisaged. The resolution of the spectrometer is very poor, attaining 70% FWHM at the lower Q values.

As can be seen, these figures represent a modest SANS spectrometer performance. Even in this case several studies on colloid particles and micelles can be performed but there are other cases, like in polymer science, the application of such a facility would be very limited, and an improvement in performance is highly desirable.

V. POSSIBLE IMPROVEMENTS ON SANS SPECTROMETER DESIGN

The most popular way to improve the performance of a SANS spectrometer, which is actually implanted in several laboratories, is to install a cold moderator in front of the entrance of the beam hole. A small volume of liquid hydrogen, cooled to temperatures around 20 K, in the process of slowing down the energy of the neutrons, enables to shift the mean energy of the neutrons to lower energies. The long wavelength neutrons are in this way increased in intensity up to a factor of 100. This is the preferred method to decrease the lower side of the momentum transfer Q without the need to increase the sample to detector distance. As can easily be seen in Fig 3 all the conditions presented there are reliable in this case and even in improvement in the resolution is possible (just

multiply the number of neutrons hitting the sample by 100 in the Fig. 3). The lower limit on the momentum transfer can reach 0.005 $Å^{-1}$ or even less.

Such devices are however extremely expensive since they run with low temperature cooling devices with hydrogen. Alternatives have to be envisaged though, and one of them is a device running with a moderator, e.g., metal hydride, cooled to the nitrogen temperature. A simple calculation shows that in this case an intensity gain factor of 10 can be achieved for the low energy part of the neutron spectrum and, again, all the cases depicted in Fig. 3 would be possible. The attainable lowest momentum transfer would be 0.007 $Å^{-1}$ with a resolution of 70%.

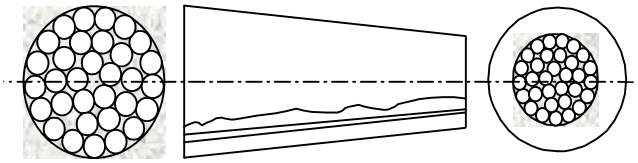


Figure 4. Stack of multiple pinhole collimators

Another possible way to improve a SANS spectrometer performance, not yet developed at the present, is the utilization of a stack of multiple pinhole collimators, depicted in Fig. 4, also called converging multi-channel collimator [2]. The motivation for such an idea is the fact that usually the neutron source area seen by the entrance aperture of a single pinhole collimator is very small compared to the available neutron source area. As in our example, R_{ns} = 2 cm whereas the beam hole radius is 15 cm. If all this area could be useful for the experiment a gain of a factor of 14 can be achieved in the neutron flux at the sample position and, again, all the cases presented in Fig. 3 could be used. It is also promising the fact that in this case the neutron rate at the sample position will not depend on the fourth power of the resolution as in the case of a simple pinhole collimator, but it depends on the square of the resolution. An understanding of the advantages of this effect is not clear at the moment and deeper analysis are now being performed.

However this system also presents not solved drawbacks. As the lengths L_1 and L_2 are changed, also R_{ns} and R_s have to change accordingly and it is nowadays an difficult technological problem to build a multi-channel collimator that enables these parameters to change satisfactorily. The second drawback is that with this system the sample diameter will be much larger, imposing that the neutron beam cross-section also enlarges. Since this beam must be transmitted by the velocity selector, it demands that the design of this device must also be adapted to wider beams. Being a mechanical device that possesses rotating disks running at high speeds, it will be difficult to build neutron velocity selectors with larger diameter disks.

VI. STACKED NEUTRON GUIDE

A neutron guide is a device that transmits neutrons of low wavelengths to large distances very efficiently. It is based on the total reflection of neutrons at very small grazing angles at suitable surfaces. Usually a neutron guide is made with rectangular shaped glass tube inside of which a coating material is deposited, e.g., Ni^{58}, that is an efficient neutron reflector. The usual aperture is ~ 15 x 5 cm and the guide is curved with a radius of curvature of several hundreds meters in order that there is no direct sight from the reactor source at the exit of the guide. The transmitted neutrons are deviated from the direct sight whereas the unwanted radiation is absorbed in the walls of the guide. By this method it is possible to obtain very clean low energy neutrons at a place with very low background radiation.

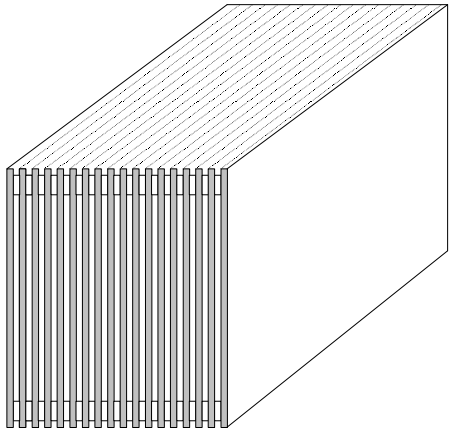


Figure 5. Sketch of a stacked neutron guide

In cases like the IEA-R1 reactor there is no possibility to install a conventional neutron guide due to lack of space. Instead, there was developed a stack of neutron guides [3] where an array of parallel guides are stacked together, as can be seen in the Fig. 5. The channel width of each guide is ~1 mm, the radius of curvature of the stack is 200 m and thus a guide only 3 meters long is enough to perform the filtering of low energy neutrons with the removal of fast neutrons and gamma radiation. Such a guide can be installed inside the reactor beam hole and thus living the rest of the space to facilities such as SANS spectrometer.

VII. CONCLUSIONS

It is shown that with the actual stage of technological development it is possible to install a modest SANS spectrometer at a beam port of the IEA-R1 reactor. With the actual reactor operating conditions and with a traditional SANS design the range of attainable momentum transfer Q would be Q_{min} = 0.01 $Å^{-1}$ to Q_{max} = 0.5 $Å^{-1}$ with the worst resolution at Q_{min} being $\Delta Q/Q$ = 0.7. A large field of systems can be studied with such a device such as

protein solutions, solutions of magnetic fluids, micelles of surfactants, formation of aggregates, etc. For systems with larger inhomogeneities like polymers, the performance would be limited.

Possibilities to improve the performance of a SANS spectrometer are also shown, in which case the lower limit of momentum transfer can be extended to 0.005 $Å^{-1}$.

ACKNOWLEDGMENTS

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